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| Application subjected to<br>Accelerated Examination   | Examiner: KONDO, Masakatsu   |
| (56) References:<br>JP 6-220494 (JP, A)<br>JP 11-50097 (JP, A)<br>JP 9-111032 (JP, A)<br>JP 5-140392 (JP, A)<br>JP 5-156292 (JP, A)<br>WO 00/56833 (WO, A1) | [0004] Issues to be Addressed by the Invention] However,<br>1,1,1,3,3-pentafluorobutane (365mfc) has a problem in that its<br>KB value (Kauri-butanol value) is approximately 14, which is<br>extremely low, and that it almost has no degreasing ability.<br>If the KB value is low, it will not be possible to exert sufficient<br>cleaning ability required of various kinds of cleaners.   |
|   | [0005] Therefore, proposals have been made to increase the<br>cleaning ability by mixing, to 1,1,1,3,3-pentafluorobutane,<br>methylene chloride or 1,1-dichloro-1-fluoroethane (HCFC-141b),<br>which have a higher KB value and degreasing-cleaning ability<br>than the above (see Publications Japanese Patent Application<br>Laid-open Publication No. 9-171185 and Japanese Patent<br>Application Laid-open Publication No. 11-152236). |

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- (54) [Title of Invention] SOLVENT COMPOSITION FOR CLEANING
- (57) [Scope of Claim]  
 [Claim 1] A solvent composition for cleaning including,  
 (a) 30 through 70 wts. of 1,1,3,3-pentafluorobutane; and  
 (b) 30 through 70 wts. of one kind of solvent or a mixed  
 solvent including at least two kinds of solvents selected from  
 among Nitromethane, nitroethane, d-limonene, and

3-methoxybutyl acetate.  
[Detailed Description of Invention]

[0001] The technical field to which the Invention Belongs] Present invention relates to a solvent composition for cleaning that contains 1,1,1,3,3-pentafluorobutane as a main ingredient and particularly relates to an art of improving the cleaning ability thereof.

[0002] [Conventional Art] Conventionally, chlorine-based solvents and fluorine-based solvents have been widely used as, for example, flux cleaners, resist removing agents, degreasing cleaners, buffering cleaners, resist removing agents, or solvents for removing adhesives. However, chlorine-based solvents are substances causing groundwater pollution, and fluorine-based solvents are substances causing ozone layer depletion; because of their environmental problems, the use thereof is becoming restricted. Therefore, there is a demand from various fields for a new solvent that would take place of the above-mentioned solvents.

[0003] 1,1,1,3,3-pentafluorobutane (365mfc, chemical formula:  $C_4H_8F_4$ ) is one such solvent (see, for example, Japanese Patent Application Laid-open Publication No. 5-171185, Japanese Patent Application Laid-open Publication No. 9-171180, Japanese Patent Application Laid-open Publication No. 6-322394, or Japanese Patent Application Laid-open Publication No. 7-188700). 1,1,1,3,3-pentafluorobutane has superior characteristics in that it does not include chlorine in its molecular structure, its ozone depletion potential (ODP) is zero, its low in toxicity, its global warming potential (GWP) is also small, and thus it is ecological and clean.

[0004] Issues to be Addressed by the Invention] However, 1,1,1,3,3-pentafluorobutane (365mfc) has a problem in that its KB value (Kauri-butanol value) is approximately 14, which is extremely low, and that it almost has no degreasing ability. If the KB value is low, it will not be possible to exert sufficient cleaning ability required of various kinds of cleaners.

[0005] Therefore, proposals have been made to increase the cleaning ability by mixing, to 1,1,1,3,3-pentafluorobutane, methylene chloride or 1,1-dichloro-1-fluoroethane (HCFC-141b), which have a higher KB value and degreasing-cleaning ability than the above (see Publications Japanese Patent Application Laid-open Publication No. 9-171185 and Japanese Patent Application Laid-open Publication No. 11-152236).

[0006] However, since methylene chloride is highly toxic, there are concerns about its negative influence on the human body during execution of work. Therefore, use thereof is not only preferable, but it is becoming harder to use. Further, 1,1-dichloro-1-fluoroethane (141b) has a high ozone depletion potential and in the future, it may be restricted under regulations.

[0007] The present invention has been made in view of such circumstances, and an object thereof is to provide a solvent composition for cleaning that contains 1,1,1,3,3-pentfluorobutane as a main ingredient and is superior in cleaning ability, and particularly, in degreasing-cleaning ability.

[0008] Means for Resolving the Issue A solvent composition for cleaning according to the present invention for achieving such cleaning in including: (a) 30 through 70 wt% of 1,1,1,3,3-pentfluorobutane; and (b) 30 through 70 wt% of one kind of solvent or a mixed solvent including at least two kinds of solvents selected from among nitromethane, nitroethane, d-limonene, and 3-methoxybutyl acetate.

[0009] [0010]

[0011]

[0012]

[0013]

[0014]

[0015]

Nitromethane, nitroethane, d-limonene, and 3-methoxybutyl acetate are such solvents found by the present inventor as to be suitable for increasing the cleaning ability of 1,1,1,3,3-pentfluorobutane. These have sufficient degreasing-cleaning ability.

[0016] 1,1,1,3,3-pentfluorobutane is set to 30 through 70 wt%, and one kind of solvent or a mixed solvent including at least two kinds of solvents selected from among nitromethane, nitroethane, d-limonene, and 3-methoxybutyl acetate is set to 30 through 70 wt% because if the content of the latter is too small, the increase in the cleaning ability is not so sufficient, and thus it is not possible to obtain a sufficient cleaning effect, whereas if the content of the latter is too large, the characteristics of nitromethane, nitroethane, d-limonene, and 3-methoxybutyl acetate will become too significant, and it will not be possible to take full advantage of the superior features of 1,1,1,3,3-pentfluorobutane.

[0018] Embodiment of the Invention An embodiment of a solvent composition for cleaning according to the present invention will be described below. The solvent composition for cleaning according to the present invention includes: (a) 30 through 70 wt% of 1,1,1,3,3-pentafluorobutane; and (b) 30 through 70 wt% of one kind of solvent or a mixed solvent including at least two kinds of solvents selected from among nitromethane, nitroethane, d-limonene, and 3-methoxybutyl acetate. Other than this, it is possible to achieve compositions that are superior in cleaning ability, as with the present invention, with the following two types of solvent compositions for cleaning.

[0019] [1] A solvent composition including: (a) 30 through 80 wt% of 1,1,1,3,3-pentafluorobutane; and (b) 20 through 70 wt% of normal propyl bromide and/or isopropyl bromide.

[0020] [2] A solvent composition including: (a) 27 through 80 wt% of 1,1,1,3,3-pentafluorobutane; (b) 20 through 70 wt% of normal propyl bromide and/or isopropyl bromide; and (c) 3 through 15 wt% of one kind of solvent or a mixed solvent including at least two kinds of solvents selected from among alkanes with a carbon number of 5 or more, cycloalkanes with a carbon number of 5 or more, or alcohols.

[0021] Normal propyl bromide (synonym: n-bromyl bromide: 1-bromo-2-propane, referred to simply as NPB) and isopropyl bromide (synonym: isopropyl bromide: 2-bromo-2-propane, referred to simply as IPB below) have been found to be solvents that are suitable for increasing the cleaning ability of 1,1,1,3,3-pentafluorobutane. NPB and IPB have a relatively high KB value of 125 and are superior in degreasing and cleaning. Further, as with 1,1,1,3,3-pentafluorobutane, they are nonflammable and have incombustible or flame-resistant characteristics and are therefore not classified as hazardous materials and are safe and easy to handle, and they have superior characteristics in that they do not include chlorine or fluorine in their molecular structures. Their ozone depletion potential (ODP) and their global warming potential (GWP) are also small, and thus they are ecological and clean. By mixing NPB and/or IPB, which are high in KB value, to 1,1,1,3,3-pentafluorobutane, it is possible to achieve a significant increase in cleaning ability, and particularly, degreasing-cleaning ability, and thus, it is possible for the solvent to exhibit sufficient performance as various types of cleaners. Further, as with

conventional cases, it is nonflammable and thus is not classified as hazardous material and is safe and easy to handle. Further, its ozone depletion potential (ODP) and global warming potential (GWP) are also small, and thus, it is ecological and clean. 1,1,1,3,3-pentafluorobutane is set to 30 through 80 wts and NPB and/or IPB is set to 20 through 70 wts because if the content of NPB and/or IPB is too small, then the cleaning ability will not be increased that much and a sufficient cleaning effect cannot be obtained. Further, if the content of NPB and/or IPB is too large, then the characteristics of NPB and/or IPB will become too significant and dissolvability will become too large, and it will not be possible to take full advantage of the superior features of 1,1,1,3,3-pentafluorobutane. By ensuring at least 30 wts in content of 1,1,1,3,3-pentafluorobutane, its characteristics can be exhibited. Further, at least one kind of solvent selected from alkanes with a carbon number of 5 or more, cycloalkanes with a carbon number of 5 or more, or alcohols is mixed in order to ① reduce the content of 1,1,1,3,3-pentafluorobutane, and ② to dissolve water-soluble inorganic substances etc. contained in flux etc. that cannot be sufficiently dissolved by normal propyl bromide and/or isopropyl bromide. 1,1,1,3,3-pentafluorobutane is extremely expensive and may give rise to a significant increase in cost if it is used in large quantities, and therefore, by mixing the solvents described above, which are much inexpensive, as a substitute, it is possible to avoid a significant increase in cost. The content of these solvents is set to 3 wts or more in order to sufficiently dissolve the water-soluble inorganic substances etc. contained in flux etc. Further, the content of these solvents is set to 15 wts or less because if too much of these solvents are mixed, the solvent composition will be turned into a combustible since these solvents are flammable and are classified as hazardous materials. As regards normal propyl bromide (NPB) and isopropyl bromide (IPB), either one may be blended individually, or both may be blended. Further, since NPB and IPB are likely to cause metal reaction with aluminum, its alloys, etc., it is preferable to add a slight amount or a small amount of at least one kind of substance selected from a group consisting of nitroalkanes, ethers, epoxides, and amines as a stabilizer for preventing such a reaction. Further, in the present cleaning solvent, it is preferable to add a slight amount or a small amount of perfume such as d-limonene to control the odor of NPB.

[0023] Similarly, as for 1,1,1,3,3-pentafluorobutane, if the object to be cleaned is made of iron, zinc, aluminum, copper,

brass, etc. when it is used for cleaning in a heated state or as steam, there are cases in which it becomes unstable because of an influence caused by the metal. Therefore, it is preferable to add, as a stabilizer, at least one kind of compound selected from nitro compounds, phenols, amines, ethers, amylenes, epoxides, or triazoles. Specifically, as stabilizers, there are: epoxides such as propylene oxide, 1,2-butylen oxide, and glycidol; phosphites such as dimethyl phosphate, diisopropyl phosphite, and diphenyl phosphite; thiophosphites such as trilauryl triphosphite; phosphine sulphides such as triphenoxypyrophosphine sulphide and trimethylphosphine sulphide; boron compounds such as boric acid, triethyl borate, triphenyl borate, phenylboronic acid, and diphenylboronic acid; phenols such as 2,6-di-tert-butyl-para-cresol; nitroalkanes such as methyl nitromethane and nitroethane; acrylic esters such as methyl acrylate and ethyl acrylate; and also, dioxane, tert-butanol, pentaerythritol, and para-isopropenyl toluene. As for the amount of addition of these stabilizers, it is preferable to set the amount to 0.01 through 5 wts of the whole amount of the solvent composition for cleaning, although the amount is not to be particularly limited.

[0024] On the other hand, it is possible to list, for example, the following as alkanes with a carbon number of 5 or more and cycloalkanes with a carbon number of 5 or more, but they are not limited to these: pentane, 2-methyl butane, 2,2-dimethyl pentane, hexane, 2-methyl pentane, 3-methyl pentane, 2,2-dimethyl butane, 2,3-dimethyl butane, heptane, 2-methyl hexane, 3-methyl hexane, 2,3-dimethyl pentane, 2,4-dimethyl pentane, octane, 2,2,3-trimethyl pentane, 2,2,4-trimethyl pentane, cyclopentane, methyl cyclopentane, cyclohexane, methyl cyclohexane, and ethyl cyclohexane. Further, alcohols include, for example: methanol, ethanol, 1-propanol, n-propanol, n-butanol, i-butanol, s-butanol, and t-butanol. As regards the alkanes with a carbon number of 5 or more, the cycloalkanes with a carbon number of 5, and the alcohols, one kind of solvent may be mixed, or a mixed solvent containing two kinds or more of them may be mixed.

[0025] Furthermore, as regards nitromethane, nitroethane, d-limonene, and 3-methoxybutyl acetate, at least one kind of solvent selected from the above may be mixed, and it is also needless to say that two kinds or more of the above may be mixed.

[0026] As main applications of the solvent composition for cleaning according to the present invention, it is possible to name: resist removing agents, flux cleaners, degreasing cleaners for oils and fats etc., buffering cleaners, solvents for

dry cleaning, removing agents for grease, oil, wax, ink etc., solvents for paint, extractants, cleaners for various articles made of glass, ceramics, rubber, metal etc. and particularly for IC parts, electrical equipments, precision equipments, optical lenses, etc., or water removing agents.

[0027] Further, as for the cleaning method to which the solvent composition for cleaning according to the present invention can be applied, there are, for example, manual wiping, immersion, spraying, shaking, ultrasonic cleaning, and steam cleaning.

[0028] Next, various tests that were carried out for confirming the performance of the solvent composition for cleaning according to the present invention are described below.

<< Machine Oil Cleaning Test >>

[0029] Test pieces made of SUS-304 (length 25 mm × width 30 mm × thickness 2 mm) were prepared, and, after immersing these test pieces into machine oil (CQ-30; made by Nippon Oil Co., Ltd.), they were immersed into each cleaning fluid for approximately 3 minutes. After subjecting the test pieces to a drying process, the cleansing state of the test pieces was studied. Cleaning fluids obtained by mixing each of normal propyl bromide (NPB), isopropyl bromide (IPB), nitroethane, d-limonene, and 3-methoxybutyl acetate (3-MBA) to 1,1,1,3-pentafluorobutane (365mfc) were used. The following table 1 through table 3 summarize the composition of each cleaning fluid and the results of cleaning. It should be noted that a small amount of nitroethane is mixed, as a stabilizer, to the cleaning fluid to which normal propyl bromide (NPB) or isopropyl bromide (IPB) is mixed.

[0030]

[Table 1]  
Machine Oil Cleaning Test 1 (365mfc - NPB)

|                  | A   | B  | C  | D  | E  | F  | G  | H  | I  | J  |
|------------------|-----|----|----|----|----|----|----|----|----|----|
| 365mfc (wt%)     | 100 | 80 | 85 | 80 | 75 | 70 | 65 | 60 | 40 |    |
| NPB (wt%)        | 0   | 10 | 15 | 20 | 25 | 30 | 35 | 40 | 50 | 60 |
| Cleaning results | x   | x  | x  | x  | x  | x  | x  | x  | x  | x  |

x: small cleaning effect O: satisfactory ⊙: extremely good  
365mfc: 1,1,1,3-pentafluorobutane (C<sub>4</sub>H<sub>8</sub>F<sub>8</sub>)  
NPB: normal propyl bromide

[Table 2]  
Machine Oil Cleaning Test 2 (365mfc - IPB)

O: satisfactory X: small cleaning effect  
365mfc: 1,1,1,3-pentafluorobutane (C<sub>4</sub>H<sub>8</sub>F<sub>8</sub>)

## 3-MBA: 3-methoxybutyl acetate

[0031] From these test results, it has been found that, in terms of cleaning machine oil, it is necessary to include, with respect to 1,1,1,3,3-pentafluorobutane (365mfc), 20 wt% or more of normal propyl bromide or isopropyl bromide, and 30 wt% or more of nitroethane, d-limonene, or 3-methoxybutyl acetate.

[0032] << Flux Cleaning Test >> In this test, flux (TAMURA P-AL-4 made by TAMURA Corporation) was applied to the whole surface of a printed wiring board for testing, and, after subjecting it to a burning process in an electric furnace at approximately 200°C for approximately 2 minutes, it was immersed into a cleaning fluid for approximately 3 minutes. Then, after subjecting the printed wiring board to a drying process, the cleansing state was examined. Cleaning fluids obtained by mixing each of normal propyl bromide (NPB), isopropyl bromide (IPB), nitromethane, nitroethane, d-limonene, and 3-methoxybutyl acetate (3-MBA) to 1,1,1,3,3-pentafluorobutane (365mfc) were used. The following table 6 summarizes the composition of each cleaning fluid and the results of cleaning. It should be noted that a small amount of nitroethane is mixed, as a stabilizer, to the cleaning fluid to which normal propyl bromide (NPB) or isopropyl bromide (IPB) is mixed.

[0033]

## [Table 4] Flux Cleaning Test 1 (365mfc - NPB)

|                  | A   | B  | C  | D  | E  | F  | G  | H  | I  | J  |
|------------------|-----|----|----|----|----|----|----|----|----|----|
| 365mfc (wt%)     | 100 | 90 | 70 | 65 | 60 | 55 | 50 | 40 | 30 |    |
| NPB (wt%)        | 0   | 10 | 20 | 30 | 35 | 40 | 45 | 50 | 60 | 70 |
| Cleaning results | x   | x  | x  | x  | O  | O  | O  | O  | O  | O  |

X: small cleaning effect O: satisfactory ⊙: extremely good  
365mfc: 1,1,1,3,3-pentafluorobutane ( $C_4H_8F_5$ )  
NPB: normal propyl bromide

## [Table 5] Flux Cleaning Test 2 (365mfc - IPB)

|                  | A   | B  | C  | D  | E  | F  | G  | H  | I  | J  |
|------------------|-----|----|----|----|----|----|----|----|----|----|
| 365mfc (wt%)     | 100 | 90 | 80 | 70 | 65 | 60 | 55 | 50 | 40 | 30 |
| Cleaning results | x   | x  | x  | x  | O  | O  | O  | O  | O  | O  |

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| IPB (wt%)        | 0 | 10 | 20 | 30 | 35 | 40 | 45 | 50 | 60 | 70 |
|------------------|---|----|----|----|----|----|----|----|----|----|
| Cleaning results | x | x  | x  | x  | O  | O  | O  | O  | O  | O  |

[Table 6] Flux Cleaning Test 3 (365mfc, nitroethane, nitromethane, d-limonene, 3-MBA)  
X: small cleaning effect O: satisfactory ⊙: extremely good  
365mfc: 1,1,1,3,3-pentafluorobutane ( $C_4H_8F_5$ )  
IPB: isopropyl bromide

|    | 365mfc (wt%) | nitroethane (wt%) | nitromethane (wt%) | d-limonene (wt%) | 3-MBA (wt%) | Cleaning ability |
|----|--------------|-------------------|--------------------|------------------|-------------|------------------|
| A  | 80           | 20                | —                  | —                | —           | x                |
| C  | 70           | 30                | —                  | —                | —           | x                |
| D  | 65           | 35                | —                  | —                | —           | ○                |
| E  | 60           | 40                | —                  | —                | —           | ○                |
| E  | 50           | 50                | —                  | —                | —           | ○                |
| F  | 40           | 60                | —                  | —                | —           | ○                |
| G  | 30           | 70                | —                  | —                | —           | ○                |
| H  | 20           | 80                | —                  | —                | —           | ○                |
| I  | 80           | —                 | 20                 | —                | —           | x                |
| J  | 70           | —                 | 30                 | —                | —           | x                |
| K  | 65           | —                 | 35                 | —                | —           | ○                |
| L  | 60           | —                 | 40                 | —                | —           | ○                |
| M  | 50           | —                 | 60                 | —                | —           | ○                |
| N  | 40           | —                 | 60                 | —                | —           | ○                |
| O  | 30           | —                 | 70                 | —                | —           | ○                |
| P  | 20           | —                 | 80                 | —                | —           | ○                |
| Q  | 80           | —                 | —                  | 20               | —           | x                |
| R  | 70           | —                 | —                  | 30               | —           | x                |
| S  | 65           | —                 | —                  | 35               | —           | x                |
| T  | 60           | —                 | —                  | 40               | —           | x                |
| U  | 50           | —                 | —                  | 50               | —           | x                |
| V  | 40           | —                 | —                  | 60               | —           | x                |
| W  | 30           | —                 | —                  | 70               | —           | x                |
| X  | 20           | —                 | —                  | 80               | —           | x                |
| Y  | 10           | —                 | —                  | 90               | —           | x                |
| Z  | 6            | —                 | —                  | 95               | —           | x                |
| AA | 80           | —                 | —                  | —                | 20          | x                |
| AB | 70           | —                 | —                  | —                | 30          | x                |
| AC | 65           | —                 | —                  | —                | 35          | x                |
| AD | 60           | —                 | —                  | —                | 40          | x                |
| AE | 50           | —                 | —                  | —                | 50          | o                |



Influence on Plastics, Rubbers, Etc. 3  
(365mfc, d-limonene, 3-MBA)

|                       | A  | B  | C  | D  | E  | F  | G  | H  | I  | J  |
|-----------------------|----|----|----|----|----|----|----|----|----|----|
| 365mfc (wt%)          | 70 | 60 | 50 | 40 | 30 | 70 | 60 | 50 | 40 | 30 |
| d-limonene (wt%)      | 30 | 40 | 50 | 60 | 70 | —  | —  | —  | —  | —  |
| 3-MBA (wt%)           | —  | —  | —  | —  | —  | 30 | 40 | 50 | 60 | 70 |
| viny chloride (soft)  | O  | O  | x  | x  | O  | x  | x  | x  | x  | x  |
| vinyl chloride (hard) | O  | O  | x  | x  | O  | x  | x  | x  | x  | x  |
| polycarbonate         | O  | O  | O  | O  | O  | O  | O  | O  | O  | O  |
| acrylic resin         | O  | O  | O  | O  | O  | O  | O  | O  | O  | O  |
| nylon 66              | O  | O  | O  | O  | O  | O  | O  | O  | O  | O  |
| polyacetal            | O  | O  | O  | O  | O  | O  | O  | O  | O  | O  |
| polyurethane resin    | O  | O  | O  | O  | O  | O  | O  | O  | O  | O  |
| phenolic resin        | O  | O  | O  | O  | O  | O  | O  | O  | O  | O  |
| epoxy resin           | O  | O  | O  | O  | O  | O  | O  | O  | O  | O  |
| melamine resin        | O  | O  | O  | O  | O  | O  | O  | O  | O  | O  |
| urea resin            | O  | O  | O  | O  | O  | O  | O  | O  | O  | O  |
| polyethylene          | O  | O  | O  | O  | O  | O  | O  | O  | O  | O  |
| polypropylene         | O  | O  | O  | O  | O  | O  | O  | O  | O  | O  |
| fluoro rubber         | O  | O  | O  | O  | O  | O  | O  | O  | O  | O  |
| chloroprene rubber    | O  | O  | O  | O  | O  | O  | O  | O  | O  | O  |
| silicone rubber       | O  | O  | O  | O  | O  | O  | O  | O  | O  | O  |
| urethane rubber       | O  | O  | O  | O  | O  | O  | O  | O  | O  | O  |
| SBR                   | O  | O  | O  | O  | O  | O  | O  | O  | O  | O  |
| natural rubber        | O  | O  | O  | O  | O  | O  | O  | O  | O  | O  |
| butyl rubber          | O  | O  | O  | O  | O  | O  | O  | O  | O  | O  |

O : no influence    X : having influence  
365mfc: 1,1,1,3,3-pentafluorobutane ( $C_4H_3F_3$ )  
3-MBA: 3-methoxybutyl acetate

[0037] From these results, it was possible to confirm that in some cases, soft and hard vinyl chloride and polycarbonate are negatively affected. It was found that, if negative influence is to be kept from being exerted on soft vinyl chloride, hard vinyl chloride, and polycarbonate, then it is necessary to set the content of normal propyl bromide and isopropyl bromide to 45 wt% or less, and set the content of nitromethane, to nitroethane, d-limonene, and 3-methoxybutyl acetate (3-MBA) to 50 wt% or less, with respect to 1,1,1,3,3-pentafluorobutane (365mfc).

[0038] << Flammability Test >>

In this test, a study was made on the relationship between flammability and the content for when 1,1,1,3,3-pentafluorobutane (365mfc) includes alkanes with a carbon number of 5 or more, cycloalkanes with a carbon number of 5 or more, or alcohols, because these solvents

have flammability and are combustibles. Ethyl alcohol (ethanol), methyl alcohol (methanol), and heptane were used as the solvents to be mixed. The following table 10 summarizes the test results. It should be noted that the flammability was studied according to the Tag closed cup method.

[0039]

[Table 10]

Flammability Test 1

|   | 365mfc | NPB    | ethanol | methanol | heptane | flammability |
|---|--------|--------|---------|----------|---------|--------------|
|   | (wt %) | (wt %) | (wt %)  | (wt %)   | (wt %)  |              |
| A | 80     | 20     | 0       | —        | —       | no           |
| B | 77     | 20     | 3       | —        | —       | no           |
| C | 76     | 20     | 5       | —        | —       | no           |
| D | 72     | 20     | 8       | —        | —       | no           |
| E | 70     | 20     | 10      | —        | —       | no           |
| F | 67     | 20     | 13      | —        | —       | no           |
| G | 66     | 20     | 16      | —        | —       | no           |
| H | 62     | 20     | 18      | —        | —       | yes          |
| I | 60     | 20     | 20      | —        | —       | yes          |
| J | 77     | 20     | —       | 3        | —       | no           |
| K | 76     | 20     | —       | 5        | —       | no           |
| L | 72     | 20     | —       | 8        | —       | no           |
| M | 70     | 20     | —       | 10       | —       | no           |
| N | 67     | 20     | —       | 13       | —       | no           |
| O | 65     | 20     | —       | 16       | —       | yes          |
| P | 62     | 20     | —       | 18       | —       | yes          |
| Q | 60     | 20     | —       | 20       | —       | yes          |
| R | 77     | 20     | —       | —        | 3       | no           |
| S | 75     | 20     | —       | 5        | —       | no           |
| T | 72     | 20     | —       | 8        | —       | no           |
| U | 70     | 20     | —       | 10       | —       | no           |
| V | 67     | 20     | —       | 13       | —       | no           |
| W | 65     | 20     | —       | 15       | —       | no           |
| X | 62     | 20     | —       | 18       | —       | yes          |
| Y | 60     | 20     | —       | 20       | —       | yes          |

365mfc: 1,1,1,3,3-pentafluorobutane ( $C_4H_3F_3$ )

NPB: normal propyl bromide

[0040] From these results, it was found that, since flammability arises when 18 wt% or more of each solvent of ethyl alcohol, methyl alcohol, and heptane is contained, in order to keep the solvent composition for cleaning (type 2) according to the present invention from becoming flammable, it is

necessary to set the content of these solvents to 15 wts or less.  
[0041] Further, since nitromethane, nitroethane,  
d-limonene, and 3-methoxybutyl acetate (3-MBA) similarly have  
flammability, the relationship between flammability and the  
content of these solvents was also studied. The following table  
11 summarizes the test results. It should be noted that the  
flammability was measured according to the Tag closed cup  
method.

[0042]

[Table 11]  
Flammability Test 2  
(365mfc, nitroethane, nitromethane, d-limonene, 3-MBA)

|    | 365mfc<br>(wt %) | nitroethane<br>(wt %) | nitromethane<br>(wt %) | d-limonene<br>(wt %) | 3-MBA<br>(wt %) | flammability |
|----|------------------|-----------------------|------------------------|----------------------|-----------------|--------------|
| A  | 70               | 30                    | —                      | —                    | —               | no           |
| B  | 60               | 40                    | —                      | —                    | —               | no           |
| C  | 50               | 50                    | —                      | —                    | —               | no           |
| D  | 40               | 60                    | —                      | —                    | —               | no           |
| E  | 30               | 70                    | —                      | —                    | —               | yes          |
| F  | 20               | 80                    | —                      | —                    | —               | —            |
| G  | 10               | 90                    | —                      | —                    | —               | —            |
| H  | 70               | —                     | 30                     | —                    | —               | no           |
| I  | 60               | —                     | 40                     | —                    | —               | no           |
| J  | 50               | —                     | 50                     | —                    | —               | no           |
| K  | 40               | —                     | 60                     | —                    | —               | no           |
| L  | 30               | —                     | 70                     | —                    | —               | no           |
| M  | 20               | —                     | 80                     | —                    | —               | no           |
| N  | 10               | —                     | 90                     | —                    | —               | yes          |
| O  | 70               | —                     | —                      | 30                   | —               | no           |
| P  | 60               | —                     | —                      | 40                   | —               | no           |
| Q  | 50               | —                     | —                      | 50                   | —               | no           |
| R  | 40               | —                     | —                      | 60                   | —               | no           |
| S  | 30               | —                     | —                      | 70                   | —               | no           |
| T  | 20               | —                     | —                      | 80                   | —               | no           |
| U  | 10               | —                     | —                      | 90                   | —               | no           |
| V  | 5                | —                     | —                      | 95                   | —               | yes          |
| W  | 70               | —                     | —                      | 30                   | no              | —            |
| Y  | 60               | —                     | —                      | 40                   | no              | —            |
| Z  | 50               | —                     | —                      | 50                   | no              | —            |
| AA | 40               | —                     | —                      | 60                   | no              | —            |
| AB | 30               | —                     | —                      | 70                   | no              | —            |
| AC | 20               | —                     | —                      | 80                   | no              | —            |
| AD | 10               | —                     | —                      | 90                   | no              | —            |
| AE | 5                | —                     | —                      | 95                   | yes             | —            |

X : corrosion O : no corrosion  
365mfc: 1,1,1,3-pentafluorobutane (C<sub>4</sub>H<sub>9</sub>F<sub>5</sub>)  
NPB: normal propyl bromide  
IPB: isopropyl bromide

| Metal Corrosion Test 1 (365mfc, NPB, IPB) |    |    |    |    |    |    |
|---|----|----|----|----|----|----|
|   | A  | B  | C  | D  | E  | F  |
| 365mfc (wt%)                              | 80 | 70 | 60 | 50 | 40 | 30 |
| NPB (wt%)                                 | 20 | 30 | 40 | 50 | 60 | 70 |
| IPB (wt%)                                 | —  | —  | —  | —  | —  | —  |
| results                                   | O  | O  | O  | O  | O  | O  |

  

| Metal Corrosion Test 2 (365mfc, nitroethane, nitromethane, d-limonene, 3-MBA) |    |    |   |   |   |   |
|---|----|----|---|---|---|---|
|   | A  | B  | C | D | E | F |
| 365mfc (wt%)  | 70 | 30 | — | — | — | — |
| nitroethane (wt%)   | 60 | 40 | — | — | — | — |
| nitromethane (wt%)  | 50 | 50 | — | — | — | — |
| d-limonene (wt%)  | 60 | 60 | — | — | — | — |
| 3-MBA (wt%)   | 60 | 60 | — | — | — | — |
| results   | O  | O  | O | O | O | O |

[Table 13]:  
Metal Corrosion Test 2  
(365mfc, nitroethane, nitromethane, d-limonene, 3-MBA)

|   |    |    |    |    |    |    |   |   |
|---|----|----|----|----|----|----|---|---|
| E | 30 | 70 | —  | —  | —  | —  | — | O |
| F | 70 | —  | 30 | —  | —  | —  | — | O |
| G | 60 | —  | 40 | —  | —  | —  | — | O |
| H | 50 | —  | 50 | —  | —  | —  | — | O |
| I | 40 | —  | 60 | —  | —  | —  | — | O |
| J | 30 | —  | 70 | —  | —  | —  | — | O |
| K | 70 | —  | —  | 30 | —  | —  | — | O |
| L | 60 | —  | —  | 40 | —  | —  | — | O |
| M | 50 | —  | —  | 50 | —  | —  | — | O |
| N | 40 | —  | —  | 60 | —  | —  | — | O |
| O | 30 | —  | —  | 70 | —  | —  | — | O |
| P | 70 | —  | —  | —  | 30 | —  | — | O |
| Q | 60 | —  | —  | —  | 40 | —  | — | O |
| R | 50 | —  | —  | —  | —  | 50 | — | O |
| S | 40 | —  | —  | —  | —  | 60 | — | O |
| T | 30 | —  | —  | —  | —  | 70 | — | O |

X : corrosion O : no corrosion

363mfc: 1,1,1,3,3-pentafluorobutane ( $C_4H_9F_5$ )

3-MBA: 3-methoxybutyl acetate

[0046] From these results, it was confirmed that none of the solvent compositions for cleaning according to the present invention has such a negative influence as to make metal corrode.

[0047] << Summary >> The following can be acknowledged by summarizing the test results described above.

(1) In order to use the solvent composition for cleaning machine oil, it is necessary to include at least 20 wt% or more of normal propyl bromide or isopropyl bromide, and at least 30 wt% or more of nitromethane, nitroethane, d-limonene, or 3-methoxybutyl acetate (3-MBA).

(2) In order to use the solvent composition for cleaning flux, it is necessary to include at least 35 wt% or more of normal propyl bromide or isopropyl bromide, and at least 40 wt% or more of nitromethane, nitroethane, d-limonene, or 3-methoxybutyl acetate (3-MBA).

(3) In case of avoiding negative influence on soft vinyl chloride, hard vinyl chloride, and polycarbonate, the content of normal propyl bromide and isopropyl bromide is kept at 45 wt% or less, and the content of nitromethane, nitroethane, d-limonene, and 3-methoxybutyl acetate (3-MBA) is kept at 50 wt% or less.

(4) In order to keep the solvent composition from becoming flammable even when alkanes with a carbon number of 5 or more, cycloalkanes with a carbon number of 5 or more, or alcohols are mixed, the content of these solvents is set to 15

wts or less.

(5) No flammability arises even when nitromethane or nitroethane is mixed at 80 wts or more, and d-limonene or 3-methoxybutyl acetate (3-MBA) is mixed within the range of content defined according to the present invention.

(6) In the solvent composition for cleaning according to the present invention, there is no fear of affecting metal such as in terms of corrosion.

[0049] In consideration of the above-described matters, a more preferable range of content for each component is as follows.

[11'] A solvent composition for cleaning including: (a) 55 through 65 wts of 1,1,1,3,3-pentafluorobutane; and (b) 35 through 45 wts of normal propyl bromide and/or isopropyl bromide.

[12'] A solvent composition for cleaning including: (a) 40 through 62 wts of 1,1,1,3,3-pentafluorobutane; (b) 35 through 45 wts of normal propyl bromide and/or isopropyl bromide; and (c) 3 through 15 wts of one kind of solvent or a mixed solvent including at least two kinds of solvents selected from among alkanes with a carbon number of 5 or more, cycloalkanes with a carbon number of 5 or more, or alcohols.

[13'] A solvent composition for cleaning including: (a) 50 through 60 wts of 1,1,1,3,3-pentafluorobutane; and (b) 40 through 50 wts of one kind of solvent or a mixed solvent including at least two kinds of solvents selected from among nitromethane, nitroethane, d-limonene, and 3-methoxybutyl acetate.

[0050] [Effect of the Invention] According to the solvent composition for cleaning of the present invention, by including nitromethane, nitroethane, d-limonene, and/or 3-methoxybutyl acetate in 1,1,1,3,3-pentafluorobutane, it is possible to greatly increase the cleaning ability, especially the degreasing-cleaning ability, and it is possible to exhibit sufficient performance in various kinds of cleaning. Further, since the solvent composition does not have flammability, it does not fall within hazardous materials; therefore, it is safe and easy to handle. Also, its ozone depletion potential (ODP) and its global warming potential (GWP) are small, and thus it is ecological and clean.

[0051] [Abstract]

[Issue] To provide a solvent composition for cleaning that

[0052]

contains 1,1,1,3,3-pentafluorobutane as a main ingredient and that is superior in cleaning ability.  
[Means for Resolution] Normal propyl bromide, isopropyl bromide, nitromethane, nitroethane, d-limonene, or 3-methoxybutyl acetate is mixed to 1,1,1,3,3-pentafluorobutane. Further, to the above, at least one kind of solvent selected from among alkanes with a carbon number of 5 or more, cycloalkanes with a carbon number of 5 or more, or alcohols is mixed.

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(58) Field of Search (Int. Cl., DB name)  
C11D 7/50  
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CA (STN)  
CAOLD (STN)  
CAPLUS (STN)  
REGISTRY (STN)



るのが好ましい。安定感として日本的にどのようなものがある。プロビレンオキシド、1, 2-ブチレンオキシド、グリジードなどのエボキシド類、ジメチルホスファトイド、ジソプロピルホスファトイド、ジエリトリチオフォスファトイドなどのオキソガラフライド類；トリメチルホスファトイド、トリクロリトリチオキシンホスファソルフライド、トリクロリトリチオフライドなどのホスファソルフライド類；トリエチルホスファトイドなどのオキソガラフライド類；トリエチルボレート、トリエニルボレート、フェニルトリエチルボレートなどのカーボン酸などのカーボン酸合成分子。

10 2, 6-ジメチル-1-ブチルパラクレゾールなどのブエーノール類；ニトロメタン、ニトロエタンなどのニトロアルカン類；アクリル酸メチル、アクリル酸エチルなどアクリル酸エステル類；その他のオキサン、トリエチルエチルエステル類；アセトニトリル、ハイドロペニトールアルケン類。これらが安定性の既知化合物については特に好ましい。

11 0.1～6重量%程度に設定するのが好ましい。

100.241 一方、炭酸ガス以上のアルカン及び既報  
数以上の中級アルカン類としては、ベンゼン、2-メチルブタン、2, 3-メチルブロバン、ヘキサン、2-ジメチルブタン、3-メチルブロバン、2, 2-ジメチルブタン、2, 3-ジメチルブロバン、ヘプタン、2-ジメチルヘキサン、3-メチルヘキサン、2, 3-ジメチルヘキサン、2, 4-ジメチルヘキサン、オクタン、2, 2, 3-トリメチルヘキサン、2, 2, 4-トリメチルヘキサン、シクロヘキサン、メチルシクロヘキサン

12 1. 1. 1. 3-ベンゼンフルオロブタンは、液体も燃え難いが、それより少しうつて大過剰供給で爆発がおきないことがから、これよりも少しつけて供給したことが可能である。1. 1. 1. 3-ベンゼンフルオロブタンの含水量を少しづつ上げて供給が可能である。これが代わりに燃焼して、大過剰供給アンプを回避することができる。これらの結果の含水量を3重量%以上としたの

| [表2] 検査油の洗浄試験1(365ml/o-NPB) |     |    |
|-----------------------------|-----|----|
|                             | A   | B  |
| 365ml/o(重量%)                | 100 | 90 |
| NPB(重量%)                    | 0   | 10 |
| 洗浄結果                        | x   | x  |

\*:洗浄効果少 O:良好  
NPB:ノルマルプロピルブロマイド

  

| [表3] 検査油の洗浄試験2(365ml/o-IPB) |     |    |
|-----------------------------|-----|----|
|                             | A   | B  |
| 365ml/o(重量%)                | 100 | 90 |
| IPB(重量%)                    | 0   | 10 |
| 洗浄結果                        | x   | x  |

\*:洗浄効果少 O:良好  
NPB:ノルマルプロピルブロマイド

[表2]

| 投紙中の洗浄試験2(365mfc-IPB) |     |    |
|-----------------------|-----|----|
|                       | A   | B  |
| 365mfc(重量%)           | 100 | 90 |
| IPB(重量%)              | 0   | 10 |
| 洗浄結果                  | x   | x  |

\* 365mfc:1, 1, 1, 3-ベンツアル  
IPB:1プロピルブロマイド

[表3]

6 m/s)に対し、ノルマルプロブロマイド(NPB)、イソプロブロブロマイド(IPB)、ニトロメタノン、ニトロエタン、d-メチルヘキサンおよび3-メチルキシアン、ジアセタセテート(3-MBA)を混合したものを使用。ブチルセラセテート(3-MBP)を混合したものを使用した。次の表1～表3は既報の結果と既往結果をまとめたものである。なお、ノルマルプロブロブロマイド(NPB)またはイソプロブロブロマイド(IPB)が混合される既往例には、安妥剤としてニトロエタンが若千量混合されている。

|    | E  | F  | G  | H  | I  | J  |
|----|----|----|----|----|----|----|
| 1) | 75 | 70 | 65 | 60 | 50 | 40 |
| 2) | 25 | 30 | 35 | 40 | 50 | 60 |
| 3) | O  | ⊕  | ⊖  | ⊖  | ⊕  | ⊕  |
| 4) |    |    |    |    |    |    |

(非燃性に良好)  
ジタラン( $C_4H_8F_4$ )

三

5m (c) )に対し、ノルマルプロピルブロマイド (NPB)、イソプロピルブロマイド (IPB)、ニトロメタシン、エトロエタン、d-メリキシノンおよびS-メチルブチルアセテート (3-MBA) を底合したものと使用した。次の表1～表3は洗浄液の組成と洗浄効果をまとめたものである。なお、ノルマルプロピルブロマイド (NPB) またはイソプロピルブロマイド (IPB) が底合される洗浄液には、安定剤としてニトロエタンが接着剤に含まれている。

|   | E  | F  | G  | H  | I  | J  |
|---|----|----|----|----|----|----|
| E | 75 | 70 | 65 | 60 | 50 | 40 |
| F | 25 | 30 | 35 | 40 | 60 | 60 |
| G | 0  | ④  | ⑥  | ⑦  | ⑧  | ⑨  |
| H | 0  | ④  | ⑥  | ⑦  | ⑧  | ⑨  |
| I | 0  | ④  | ⑥  | ⑦  | ⑧  | ⑨  |
| J | 0  | ④  | ⑥  | ⑦  | ⑧  | ⑨  |

表1  
通常に使用する  
溶剤の組成





[0039] ノール)、ヘブタンを使用した。燃焼結果を次の表10にまとめた。なお、引火性はタクセメント方式により調べた。

[0040] 引火性試験1(365mls-NPB、エチルメタノール、ヘブタン)

| 365mls<br>(重量%)                                  | NPB<br>(重量%) | エチルメタノール<br>(重量%) | ヘブタン<br>(重量%) | 引火性 |
|--|--------------|-------------------|---------------|-----|
| A 80   | 20           | 0                 | -             | -   |
| B 77   | 20           | 3                 | -             | -   |
| C 75   | 20           | 6                 | -             | -   |
| D 72   | 20           | 8                 | -             | -   |
| E 70   | 20           | 10                | -             | -   |
| F 67   | 20           | 13                | -             | -   |
| G 65   | 20           | 15                | -             | -   |
| H 62   | 20           | 18                | -             | -   |
| I 60   | 20           | 20                | -             | -   |
| J 77   | 20           | -                 | 3             | -   |
| K 75   | 20           | -                 | 6             | -   |
| L 72   | 20           | -                 | 8             | -   |
| M 70   | 20           | -                 | 10            | -   |
| N 67   | 20           | -                 | 13            | -   |
| O 65   | 20           | -                 | 15            | -   |
| P 62   | 20           | -                 | 18            | -   |
| Q 60   | 20           | -                 | 20            | -   |
| R 77   | 20           | -                 | -             | 有   |
| S 75   | 20           | -                 | -             | 有   |
| T 72   | 20           | -                 | -             | 有   |
| U 70   | 20           | -                 | -             | 有   |
| V 67   | 20           | -                 | -             | 有   |
| W 65   | 20           | -                 | -             | 有   |
| X 62   | 20           | -                 | -             | 有   |
| Y 60   | 20           | -                 | -             | 有   |
| 365mls-1,1,3,3-ベンツアリルプロピルアセテート-NPB-ノルマルプロピルブロマイド |              |                   |               |     |

[0041] NPB-ノルマルプロピルブロマイド

365mls-1,1,3,3-ベンツアリルプロピルブロマイド

365mls-1,1,3,3-ベンツアリルプロピルブロマイド

365mls-1,1,3,3-ベンツアリルブロマイド

[0042] この結果から、エチルアルコール、メチルアルコール及び3メトキシ・ブチルアセテート(3-MBA)についても同様に引火性があることから、これらを含有すると引火性を持つようになっていることから、未の説明について述べた。此結果を次の表11にまとめた。なお、引火性はタグ燃焼方式により測定した。

[0042]

[表11]

[0043] この結果から、ニトロミン及びニトロエタンについては8.0重量%以上、またd-リモネン及びd-リモネン-3-メトキシ・ブチルアセテート(3-MBA)についでは9.5重量%以上でそれぞれ引火性を持つようになつてあるが、本項に供する燃焼用試験装置(タイプ3)において引火性を検出しないようにするためにニトロメタン及び3-メトキシ・ブチルアセテート(3-MBA)の含有量を7.0重量%以下に設定した。

[0043]

[表12]

[0044] (金属容器試験) この結果では、JIS-K1600に従つて、燃焼後の液滴の液滴部および残部の各々にアルミニウム片(A1100P)を配置し、約4時間後の金属の目詮状況を観察した。

[0044]

[表12]

| 365mls<br>(重量%) | A  | B  | C  | D  | E  | F  | G  | H  | I  | J  | K  | L  |
|-----------------|----|----|----|----|----|----|----|----|----|----|----|----|
| NPB(重量%)        | 80 | 70 | 60 | 50 | 40 | 30 | 20 | 10 | 60 | 50 | 40 | 30 |
| IPB(重量%)        | 20 | 30 | 40 | 50 | 60 | 70 | -  | -  | -  | -  | -  | -  |
| 糊類              | 0  | 0  | 0  | 0  | 0  | 0  | 0  | 0  | 0  | 0  | 0  | 0  |

X:燃焼引  
O:燃焼不  
365mls-1,1,3,3-ベンツアリルプロピルブロマイド  
NPB:ノルマルプロピルブロマイド  
IPB:イソプロピルブロマイド

[表13]



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